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Thermoluminescence and Optical Bleaching in Minerals Exhibiting Second Order Kinetics and Other Charge Retrapping Characteristics

Technical Report

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EXECUTIVE SUMMARY

The current emphasis on placing radioactive waste repositories in hard rock has created a growing need for radiation damage information on granite, basalt, shales, clays and similar materials. In contrast to the information on rock salt, radiation damage information on hard rock is almost completely lacking. A few years ago, information on damage in rock salt was limited, especially above room temperature. However, extensive information on defects in the alkali halides was available and the techniques for studying damage in rock salt were fully developed. Consequently, the currently available information on radiation damage in rock salt was obtained at a rapid rate. Still, it is important to emphasize that all of the radiation damage information needed to evaluate repository selection, design, safety assessment, etc, in rock salt is not available. For example, adequate dose rate data on damage formation in salt does not exist.

The acquisition of radiation damage data for hard rock is exacerbated by a lack of established techniques for studying radiation damage in these materials, particularly on the atomic and/or crystal lattice scale. For example, most hard rock minerals are opaque and cannot be studied by the optical absorption techniques which work so well for rock salt. Thus, of necessity, our current program emphasizes the development of damage measurement techniques in granite, basalt, etc.

One technique being evaluated is based on the phenomena called thermoluminescence (which will be abbreviated TL). TL is the light emitted by a substance when it is heated and which results from prior exposure to radiation. The resulting curve of emission intensity vs. sample temperature is called a glow curve. To give just one example of the use of TL for damage studies, the TL glow curve intensity, when measured under proper conditions, is a measure of the number of defects in the sample. The feldspars, which are a major constituent of granite, are strong TL emitters. To make meaningful TL radiation damage measurements on feldspars, as well as other TL emitters, one must establish the TL kinetics. Some years ago we demonstrated, at BNL, that the feldspar albite exhibits second order TL kinetics. Recently, other laboratories have shown that the TL from deep-sea sediments, which contain feldspars, is drastically modified by optical bleaching prior to TL measurement. These observations suggest that extensive charge retrapping occurs during the TL emission process. Almost all thermoluminescent materials studied to date exhibit first order kinetics, which occurs when retrapping is negligible.

Consequently, the study described in this report was initiated to determine if the experimental TL and optical bleaching data could be understood on the basis that both processes involved extensive charge retrapping. The study consists of five parts: 1) an outline of the experimental data; 2) derivations of the commonly used first and second order TL "glow curve" expressions with emphasis on an examination of a) the approximations used in their formulation and b) their dependence on the retrapping parameters; 3) examples showing how glow curves are modified when the usual approximations do not apply; 4) the derivation of equations, for a single glow peak, showing how

the TL intensity is modified by prior optical bleaching; and 5) an extension of this formalism for materials which exhibit more than one glow peak.

The completed study appears to "explain," at least qualitatively, the existing TL and optical bleaching data. It is likely that additional TL features, not described here, can be explained by an extension of the method used. Most importantly, however, the results are required for the evaluation of TL techniques for the study of radiation damage in hard rock containing feldspars, the use of TL as a dosimeter for in situ repository measurements, the dating of crack fillings in the rock at repository and nuclear reactor sites, and similar investigations. Lastly, the results are immediately applicable to the dating of archaeological artifacts and a very large variety of minerals.

Most of this study was contained in an invited paper presented at the 2nd International Specialist Seminar on Thermoluminescence Dating, Oxford, England, September 1-6, 1980. It will be included in the conference proceedings, to be published in PACT-J. of the European Study Group on Physical, Chemical and Mathematical Techniques Applied to Archaeology. The methods outlined in this paper have been applied to a number of additional TL problems and others are under consideration. Eventually the entire formulation will be included in an expanded paper.

THERMOLUMINESCENCE AND OPTICAL BLEACHING IN MINERALS EXHIBITING SECOND ORDER KINETICS AND OTHER CHARGE RETRAPPING CHARACTERISTICS*

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ABSTRACT

Recent studies indicate that extensive charge retrapping contributes to thermoluminescence and optical bleaching mechanisms in feldspars, ocean sediments, etc. An analysis has been developed to describe individual thermoluminescence glow peaks when retrapping occurs and when the approximations used to derive the well-known first and second order kinetic expressions do not apply. It demonstrates that the glow peak shape, peak temperature, etc. depend strongly on the ratio of the charge retrapping and recombination cross sections and the ratio of the trapped electron and hole concentrations. The analysis also describes the effects induced by optical bleaching prior to thermoluminescence measurement.

I. INTRODUCTION

For more than forty years it has been known that the physical processes producing thermoluminescence in most solids can be divided into two categories. In one retrapping of the thermally released charges contributing to thermoluminescence is negligible. In this case the thermoluminescence kinetics is usually called first order. In the second category the thermally released charges undergo retrapping to an extent that extends from near negligible to extensive. When retrapping cannot be neglected the thermoluminescence kinetics is usually called second order. With surprisingly few exceptions, most thermoluminescent materials investigated in basic solid state physics studies, geoscience, archaeometric, dosimetry and other applications appears to exhibit first order kinetics, i.e. there was little reason to conclude that charge retrapping was important. However, in recent years evidence has been accumulating which indicates that the feldspars, a very ubiquitous mineral, exhibit second order thermoluminescence kinetics.

The sections that follow will contain, first, an outline of the experimental evidence for charge retrapping in certain thermoluminescence (abbreviated TL) systems. Second, the well-known expressions that describe the

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shape of the first and second order TL glow peaks, originally given by Randall and Wilkins (1945), will be derived. This will be done in a way which: 1) emphasizes the assumptions used in their derivation and 2) demonstrates the changes which occur in the glow peak shapes when these assumptions do not apply or are only approximated. Stated alternatively, a formalism will be developed to describe glow peak shapes which is slightly more general than that of Randall and Wilkins. Lastly, this formalism will be extended to describe the effects observed when materials are subjected to optical, i.e. light induced, bleaching prior to TL measurements.

II. EVIDENCE FOR RETRAPPING IN TL MEASUREMENTS

The indications that charge retrapping is an important part of the TL producing process in a number of minerals can be divided into three categories.

Irradiation and Heat Treatment Dependent Glow Peak Temperature Shifts: A careful study of the mineral albite, a "corner" member of the feldspar ternary phase diagram, using the "3-D" apparatus for determining the emission spectra during a TL measurement, showed that the maximum temperature of several glow peaks decreased as the pre-measurement dose was increased (Pasternack et al. 1976, 1977a, 1977b, 1978). The shift in peak temperature was also observed in a series of marine sediment samples which were exposed to heat treatments of increasing duration at a fixed temperature and subsequent cooling to room temperature prior to the TL glow curve being recorded (Wintle and Huntley, 1979, 1980).

Superposition of Glow Curves at High Temperatures: Both the albite study and the marine sediment studies cited above contain glow curves that coincide at temperatures beyond the peak temperatures, where the emission intensity is approaching zero. These observations can be expressed in a different way. As the area under the glow curve is changed, either by irradiating the sample or by "draining" a part of the trapped charge by heat treatment, the peak temperatures shift and the degree of glow curve superposition changes in a way which depends on the fraction of trapping centers that contain trapped charge when the TL measurement is started.

Glow Curve Changes Induced by Optical Bleaching Prior to Measurement: The marine sediment studies (Wintle and Huntley, 1979, 1980) describe glow curve measurements on a series of samples that had been subjected to optical bleaching, by simulated sunlight, for increasing periods prior to measurement. The glow curves they obtained exhibit six significant features. 1) The glow curves obtained from samples not exposed to light contain glow peaks at roughly 220C, 285C and, most likely, additional peaks at higher temperatures. As the bleaching time increases 2) the original glow peaks all decrease in intensity and 3) one, or possibly more, glow peaks appear in the 75 to 150C region which was originally devoid of peaks. 4) The light induced peaks first increase and then decrease in intensity as the bleaching

time is increased. However, 5) the total area under the glow curve decreases as the bleaching increases. Finally, 6) all observed glow peaks decrease as the bleaching time becomes large. There is a suggestion, but not a clear demonstration, that the glow peak maxima shift to higher temperatures as the total area decreases. These bleaching studies strongly suggest that the incident light has untrapped charges from deep traps, associated with high temperature glow peaks, and transferred them to shallow traps, associated with low temperature peaks. Most likely, the charge rearrangement occurs in a straightforward way. The incident light excites electrons into the conduction band where they move about freely. The mobile electrons are either retrapped in trapping centers or undergo light producing or nonradiative recombination with opposite sign charges.

As will be demonstrated below, the peak shifts, the superposition, and the bleaching effects are all consistent with the assumption that retrapping cannot be neglected in these materials.

III. EFFECTS OF RETRAPPING ON GLOW CURVE PROPERTIES

To demonstrate how retrapping of the thermally released charges affects the glow curve properties, only the simplest possible configuration will be used. Thus, consider a material containing one type of electron trap and one type of hole trap. Also, light is emitted when the electrons, thermally released from the electron traps and which have not been retrapped, recombine with trapped holes. Let N_1 be the concentration (concentration will be abbreviated conc) of electron traps, n_1 the conc of trapped electrons at time t, n_{01} the value of n_1 at t = 0, i.e. the conc of trapped electrons when the measurements are started, n_r the conc of trapped holes at time t and n_{ro} the hole conc at t = 0.

Thermal release of trapped charge: As usual the number of electrons thermally excited to the conduction band, per unit time, is given by $n_1 s_1 \exp(-E_1/kT)$ where n_1 is defined above, s_1 is the preexponential or "attempt-to-escape" frequency, E_1 the activation energy for thermal untrapping, k is Boltzmann's constant and T the temperature. (T is in degrees Kelvin for all computations but in degrees Celsius for all illustrations.)

Processes involving electrons in the conduction band: Electrons in the conduction band are subject to several competing processes. In the simple case under consideration, conduction band electrons can be retrapped in empty traps or can recombine with trapped holes. The probability that a conduction band electron will be retrapped is proportional to $\sigma_{t1}(N_1-n_1)$ where σ_{t1} is the cross-section for retrapping and (N_1-n_1) is the concentration of empty electron traps. The probability that it will undergo recombination is $\sigma_r n_r$, where σ_r is the cross section for recombination and n_r the trapped hole concentration. The fraction of thermally released electrons undergoing recombination is $(\sigma_r n_r)/[\sigma_{t1}(N_1-n_1) + \sigma_r n_r]$ and the fraction retrapped is $\sigma_{t1}(N_1-n_1)/[\sigma_{t1}(N_1-n_1) + \sigma_r n_r]$. The factor $[\sigma_{t1}(N_1-n_1) + \sigma_r n_r]$ is proportional to the total number of processes

occurring per unit time. This approach to electron and hole trapping is not recent, e.g. see Levy (1960).

The general TL glow curve expressions: Using the definitions given above, the differential equation for the trapped electron concentration, as a function of time, during a TL measurement can be written as

$$\frac{dn_1}{dt} = (-1)[n_1 s_1 \exp(-E_1/kT)]\left(1 - \frac{\sigma_{t1}(N_1-n_1)}{\sigma_{t1}(N_1-n_1)+\sigma_r n_r}\right). \quad (1)$$

The (-1) factor occurs because the trapped charge concentration is decreasing. The second factor is the concentration of trapped charge released to the conduction band, per unit time. The third factor is the fraction of released electrons that are not retrapped, i.e. the fraction that undergoes recombination. Note that in this and all similar equations the σ_{ti} and σ_r cross sections can always be expressed as the ratio σ_r/σ_{ti}. The corresponding equation for trapped holes is

$$\frac{dn_r}{dt} = (-1)[n_1 s_1 \exp(-E_1/kT)]\left(\frac{\sigma_r n_r}{\sigma_{t1}(N_1-n_1)+\sigma_r n_r}\right). \quad (2)$$

In (2) the third factor is the fraction of released electrons that recombine with trapped holes. Note that equations (1) and (2) are identical! These equations describe the trapped charge concentrations. The corresponding TL light intensity I(t) is given by

$$I(t) = -dn_1/dt \quad \text{or} \quad I(t) = -dn_r/dt. \quad (3)$$

The usual glow curve expressions: If, in (1) or (2), the probability for recombination is very much larger than the probability for retrapping, i.e. if $\sigma_r n_r >> \sigma_{t1}(N_1-n_1)$, then

$$1 - \frac{\sigma_{t1}(N_1-n_1)}{\sigma_{t1}(N_1-n_1)+\sigma_r n_r} \simeq 1 \quad (4)$$

and

$$\frac{\sigma_r n_r}{\sigma_{t1}(N_1-n_1)+\sigma_r n_r} \simeq 1. \quad (5)$$

In this situation

$$I(t) = -\frac{dn_1}{dt} = n_1 s_1 \exp(-E_1/kT). \quad (6)$$

Since $n_1 \simeq n_r$ it is likely that $\sigma_r n_r >> \sigma_{t1}(N_1-n_1)$ occurs when $\sigma_r >> \sigma_{t1}$. The equation for dn_r/dt is identical to eq. (6).

When (6) is solved using the usual condition for the sample temperature increasing linearly, i.e. $T = T_o + \beta t$ where β is the heating rate and T_o the

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initial temperature, one obtains what is usually called the first order glow curve expression (Randall and Wilkins, 1945; Garlick, 1949; and Curie, 1963).

$$I(t) = n_0 s_1 \exp(-E_1/kT) \exp\left(-\int_0^T \frac{s_1}{\beta} \exp(-E_1/kT)\,dt\right). \quad (7)$$

If both of the two approximations, $n_1 = n_r$ and $\sigma_{t1} = \sigma_r$ are inserted into (1) or (2) one obtains

$$I(t) = -\frac{dn_1}{dt} = \frac{n_1^2}{N_1} s_1 \exp(-E_1/kT). \quad (8)$$

When (8) is solved using the linear heating rate expression, $T = T_0 + \beta t$, one obtains, what is usually called, the second order glow curve expression,

$$I(t) = \frac{n_{01}^2 s_1 \exp(-E_1/kT)}{N_1\left(1 + \frac{n_{01}}{N_1}\int_0^T \frac{s_1}{\beta} \exp(-E_1/kT)\,dT\right)^2}. \quad (9)$$

The properties of (7) and (9), which have been known for 40 years or more, are summarized in Fig. 1. The first order glow peak shape and peak temperature are independent of peak area. As pointed out previously (Levy, 1978, 1979), a shift in glow peak temperature with dose, which reflects the fraction of traps that are filled when the TL measurement is started, i.e. n_{01}/N_1, indicates that the material exhibits second order kinetics. Also, Fig. 1 shows that the second order glow curves coincide as they approach low intensity on the high temperature side of the glow peak. This can be regarded as a second glow curve property indicating that retrapping is occurring in the TL process.

Glow peak properties in cases when the trapping and recombination cross sections are not equal: The dependence of glow peak shape, peak temperature and other properties in cases when the approximations used to derive the usual expressions, (7) and (9), for first and second kinetics are not fulfilled is shown in Figures 2 through 4. In these figures the usual expressions are marked by special symbols.

To illustrate the glow curve behavior obtained when $\sigma_r \neq \sigma_{t1}$, the system of equations (1), (2), and (3) has been solved using computerized numerical techniques. Figures 2 and 3 show the computed glow curves for a case where all parameters are fixed but the recombination to retrapping cross section ratio. In other words computed curves are shown for different values of the σ_r/σ_{t1} ratio. The curves marked "First" and "Second" order were computed from the usual expressions, equations (7) and (9). All of the computed glow curves have the same area. As σ_r becomes larger with respect to σ_{t1} the curves more closely resemble the first order curve which corresponds to the case of σ_r becoming infinitely large. Figure 3 demonstrates that the usual second order expression is a good approximation only when $n_1 = n_r$ and $\sigma_r = \sigma_{t1}$.

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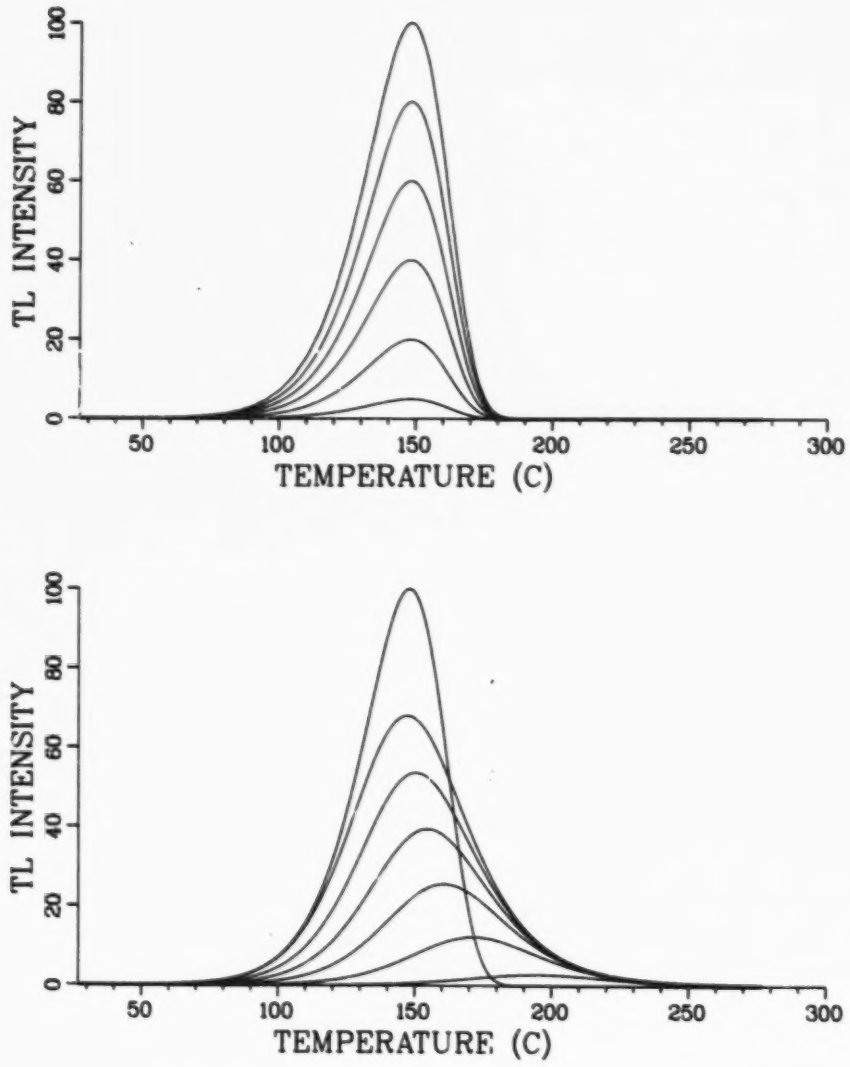

Fig. 1:- The upper group of curves was computed from the well-known first order kinetic expression, eq. (7) in the text, with initial trapped charge concentrations of 1.0, 0.8, 0.6, 0.4, 0.2, and $0.05 \times 10^{16}/cm^3$. The lower group shows the 1.0×10^{16} first order curve and curves computed from the usual second order kinetic expression, eq. (9), with 1.0, 0.8, 0.6, 0.4, 0.2, and 0.05 of the traps filled prior to the heating. The constants $E = 1.0$ eV, $s = 10^{10}$/sec and $\beta = 10C$/min were used for both. The peak temperature and shape of the first order curves are independent of initial charge concentration. These characteristics of the second order curves depend on the initial charge. Also, the second order curves coincide at high temperatures.

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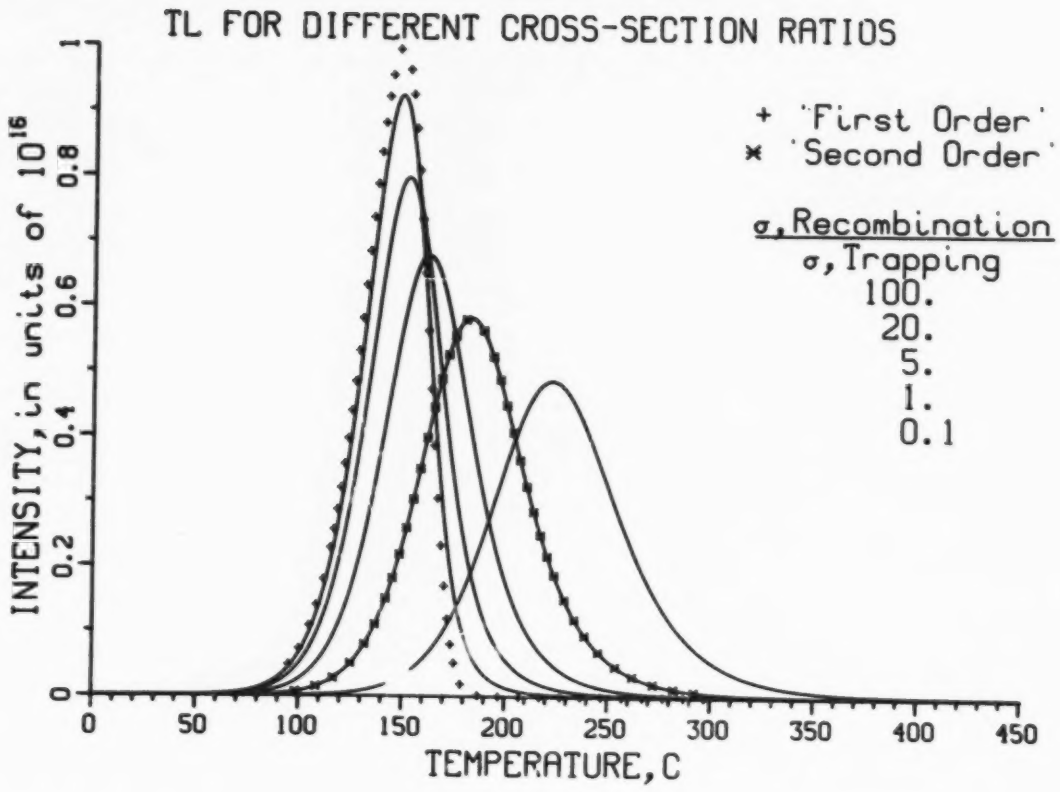

Fig. 2:- Glow curves numerically computed from the general equations (1), (2) and (3) in the text, using the parameters $N_1 = 10^{16}$ traps/cm^3, $n_{o1} = n_{ro} = 10^{15}$ charges/cm^3, $E_1 = 1.0$ eV, $s_1 = 10^{10}$/sec, $T_o = 0$ C, $\beta = 10$ C/min and the indicated σ_r/σ_{t1} ratios. The "first" and "second" order curves were computed from the usual expressions, eqs. (7) and (9). In this and similar figures the curves occur, from top to bottom, in the same order as the table of parameters.

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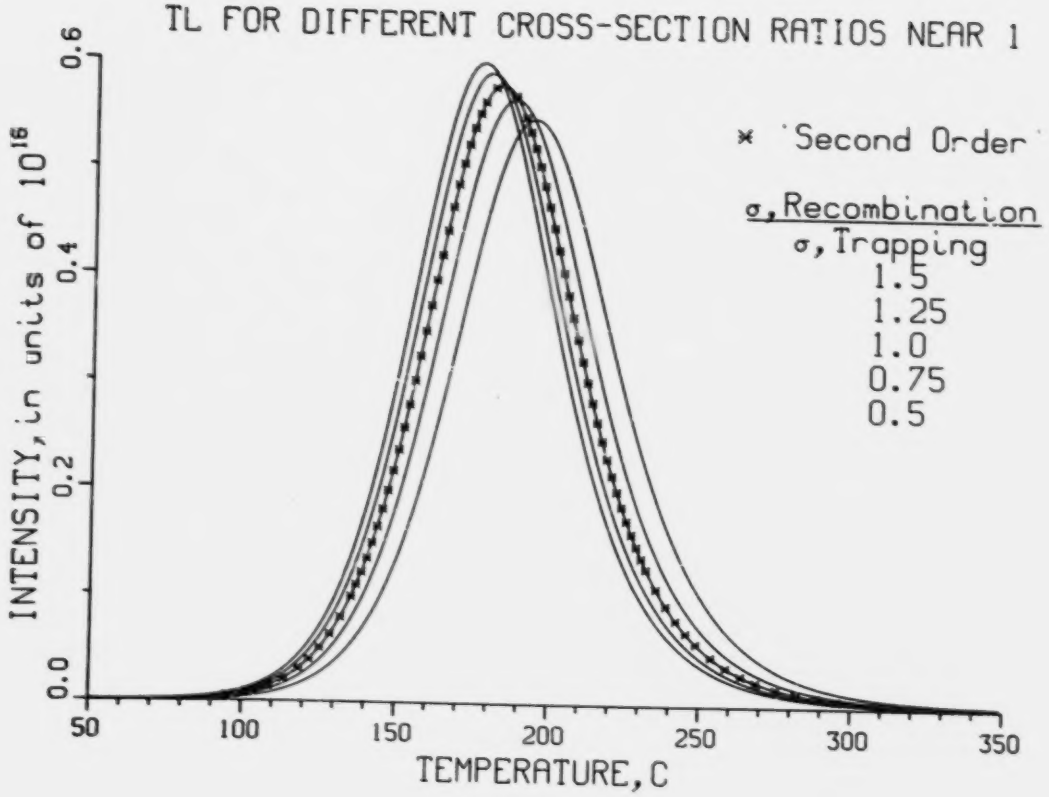

Fig. 3:- Glow curves computed with the same parameters used for Fig. 2 except that the σ_r/σ_{t1} ratios are near one. Figures 2 and 3 demonstrate that the glow curve characteristics are sensitive functions of the σ_r/σ_{t1} ratio and that first order kinetics are obtained when this ratio becomes very large.

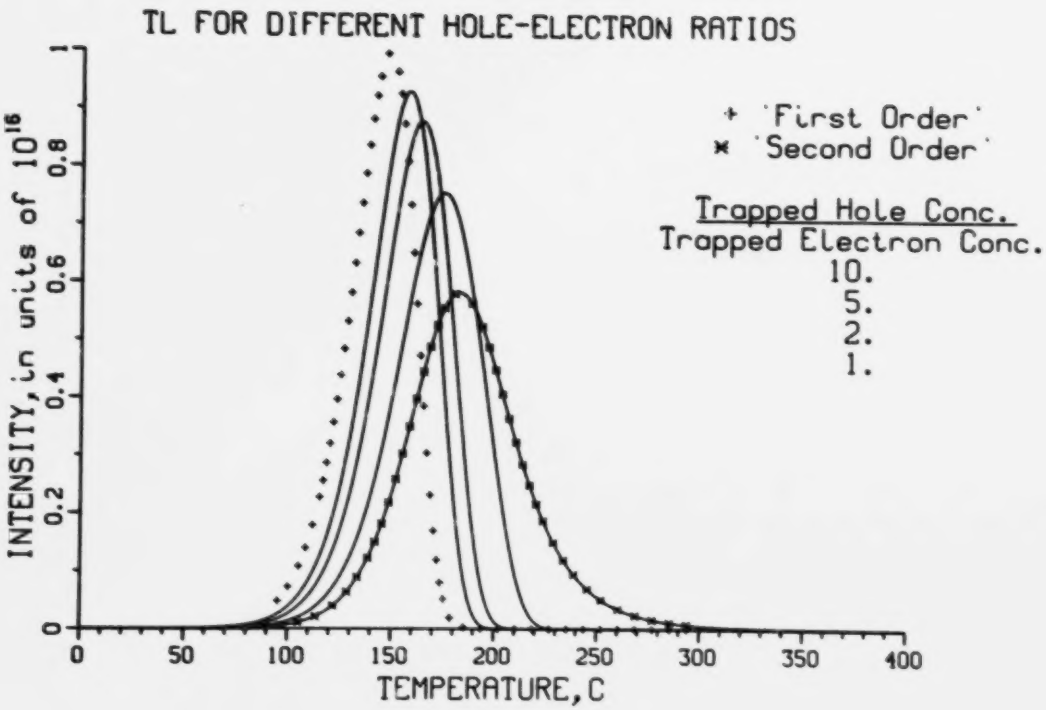

Fig. 4:- Glow curves numerically computed from the eqs. (1), (2) and (3) using the parameters $N_1 = 10^{16}$ traps/cm^3, $n_{o1} = 10^{15}$ charges/cm^3, $n_{ro} = 1.0$, 2.0, 5.0 and 10.0×10^{15} charges/cm^3, $E_1 = 1.0$ eV, $s_1 = 10^{10}$/sec, $T_o = 0$ C, $\beta = 10$ C/min and $\sigma_r/\sigma_{t1} = 1.0$. The "first" and "second" order curves were computed from the usual expressions.

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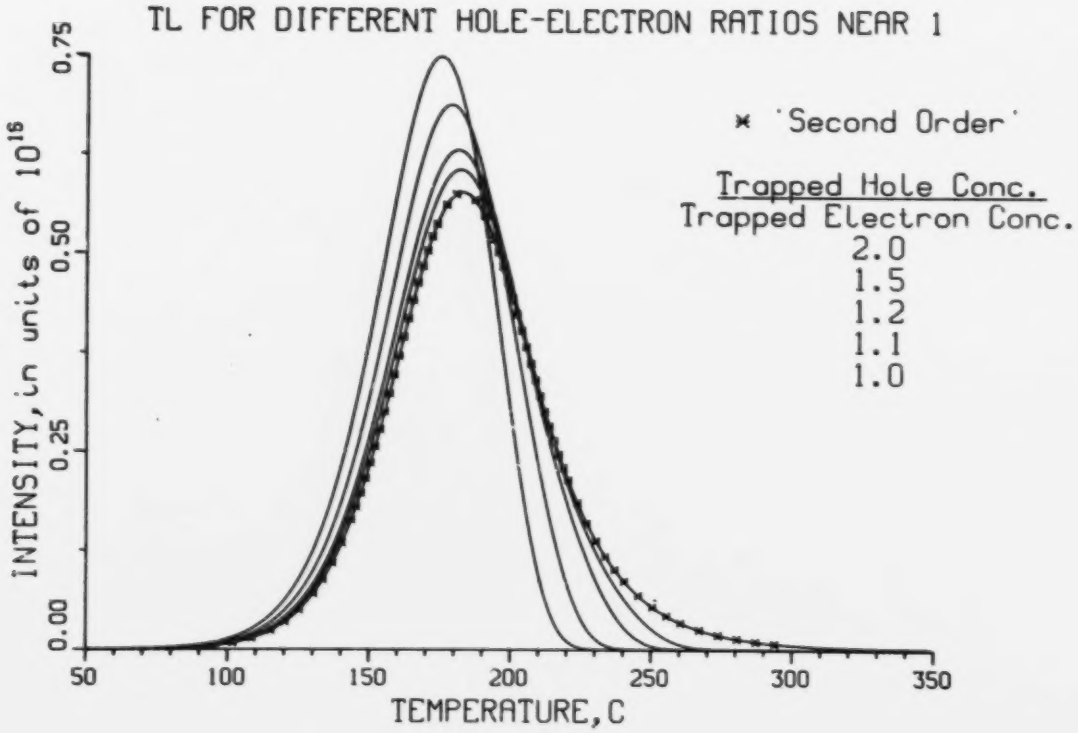

Fig. 5:- Glow curves numerically computed with the same parameters used for Fig. 4 except that n_{ro} = 1.0, 1.1, 1.2, 1.5 and 2.0×10^{15} charges/cm^3. Figures 4 and 5 demonstrate that the glow curve characteristics are sensitive functions of the n_{ro}/n_{ol} ratio and that the glow curves approach the first order shape as this ratio becomes large.

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To demonstrate that the shape of the glow peaks depends on the value or n_r, i.e. to illustrate how the shape of the peak changes when the assumption $n_1 \neq n_r$ is not fulfilled, the curves in Figures (4) and (5) were computed. In these two cases all of the constants remain fixed except n_{ro}. It is likely that the condition $n_{o1} \neq n_{ro}$ or $n_1 \neq n_r$ applies to most materials exhibiting "second-order" kinetics that contain more than one glow peak. Consider a TL emitter with two or more different types of electron traps and only one type of hole trap. Natural or laboratory irradiation will cause electrons to be trapped in the two types of electron traps and after irradiation the total number of trapped electrons will be very close to the total number of trapped holes, to maintain charge conservation. When the sample is heated, at temperatures where the lowest temperature glow curve occurs, the thermally released electrons can recombine with a trapped hole population which is equal to the sum of all trapped electrons. In other words, the total number of electrons in the lowest temperature traps can recombine with a much larger population of trapped holes. In fact, if there is only one type of hole trap and there is more than one glow peak, the trapped electron and hole populations contributing to each peak will be equal only for the highest temperature peak.

These figures also demonstrate that the usual expressions are obtained when either $\sigma_r >> \sigma_{t1}$, or $n_r >> n_1$ or both of these conditions apply. Because of space limitations, materials containing more than one type of hole trap will not be discussed.

IV. EFFECTS OF OPTICAL BLEACHING, PRIOR TO THERMOLUMINESCENCE MEASUREMENT, ON MATERIALS EXHIBITING ONE GLOW PEAK

To obtain an understanding of the effects produced by subjecting samples to optical, or light induced, bleaching prior to TL measurements, consider first the simplest possible system. Namely, the one described above consisting of N_1 electron traps, containing n_{o1} electrons at $t = 0$, n_1 electrons at time t, n_{ro} trapped holes at $t = 0$ and n_r trapped holes at time t. Also, consider that only electrons are thermally untrapped. To encompass a reasonably large range of different kinds of bleaching behavior, two of many possible bleaching mechanisms will be described. The first, in which the bleaching rate is proportional to the trapped electron concentration, has a high probability of being the mechanism actually operating in most materials. Also, this mechanism will cause bleaching to proceed at the "slowest" extreme considered. The second mechanism is based on the assumption that the photon induced charge untrapping proceeds at a constant rate. It is included to illustrate a case in which bleaching proceeds at the "fastest" extreme.

Case I--photon induced untrapping proportional to trapped charge concentration: Let ϕ be the photon flux incident on a sample. Then, in the simple case being considered, the number of trapped electrons elevated to the conduction band, per unit time and volume, is given by $\phi n_1 \sigma_{u1}$, where n_1, as defined above, is the trapped charge concentration and σ_{u1} is the cross section for photon induced untrapping. Then the equation for the trapped charge concentration as a function of time after bleaching has commenced is

$$\frac{dn_1}{dt} = (-1)(\phi n_1 \sigma_{u1})\left[1 - \frac{\sigma_{t1}(N_1 - n_1)}{\sigma_{t1}(N_1 - n_1) + \sigma_r n_r}\right] . \qquad (10)$$

This is equation (1) with the $n_1 s_1 \exp(-E/kT)$ term replaced by the $\phi n_1 \sigma_{u1}$ term. In other words, the basic equation describing optical bleaching is the same as the basic equation describing TL glow curves, but with one important difference. During a TL measurement electrons are elevated to the conduction band by thermal untrapping while during bleaching the electrons are elevated by photon induced untrapping.

The solution of (10) gives the trapped charge concentration, n_1, as a function of time after bleaching has commenced. In the case where $n_1 = n_{o1}$ at $t = 0$ and $n_1 = n_r$ it is

$$t = \frac{1}{(\phi \sigma_{u1})}\left[\frac{\sigma_{t1}}{\sigma_r} N_1 \left(\frac{1}{n_1} - \frac{1}{n_{o1}}\right) + \left(\frac{\sigma_{t1}}{\sigma_r} - 1\right) \ell n \frac{n_1}{n_{01}}\right] \qquad (11)$$

Note that, from (10), the rate that electrons are elevated to the conduction band when bleaching commences at $t = 0$ is $\phi n_{o1} \sigma_{u1}$.

Case II--photon induced untrapping at a constant rate: It is possible, but not very likely, that light incident on a sample will elevate trapped charges to the conduction band at a constant rate. For example, this could occur if the incident light produces, at a constant rate, a species, such as an exciton, which "wanders about" in the material until it interacts with trapped charges in a way that elevates them to the conduction band. In this case the number of trapped electrons elevated to the conduction band, per unit time and volume, is $\phi N_c \sigma_c$, where N_c is the concentration of crystal species, having cross section σ_c, which interacts with the photon flux, ϕ, to produce, in an unspecified way, an unchanging flow of trapped charge to the conduction band. In this case the equation for trapped charge concentration as a function of time after bleaching has commenced is

$$\frac{dn_1}{dt} = (-1)(\phi N_c \sigma_c)\left[1 - \frac{\sigma_{t1}(N_1 - n_1)}{\sigma_{t1}(N_1 - n_1) + \sigma_r n_r}\right] . \qquad (12)$$

This is equation (1) with the $n_1 s_1 \exp(-E/kt)$ term replaced, in this case by the $\phi N_c \sigma_c$ term.

The solution of (12), with $n_1 = n_{o1}$ at $t = 0$ and $n_1 = n_r$ is

$$t = \frac{1}{\phi N_c \sigma_c}\left[\frac{\sigma_{t1}}{\sigma_r} N_1 \ell n \frac{n_{o1}}{n_1} + \left(1 - \frac{\sigma_{t1}}{\sigma_r}\right)(n_{o1} - n_1)\right] . \qquad (13)$$

A number of useful results are obtained from (11) and (13). First, in both cases n_1 goes to zero only as t approaches infinity. Thus, unless some mechanism is operating other than those considered here, the trapped charge cannot be reduced to zero by optical bleaching alone. However, by sufficiently long bleaching it can be reduced to a chosen low level. Second, a

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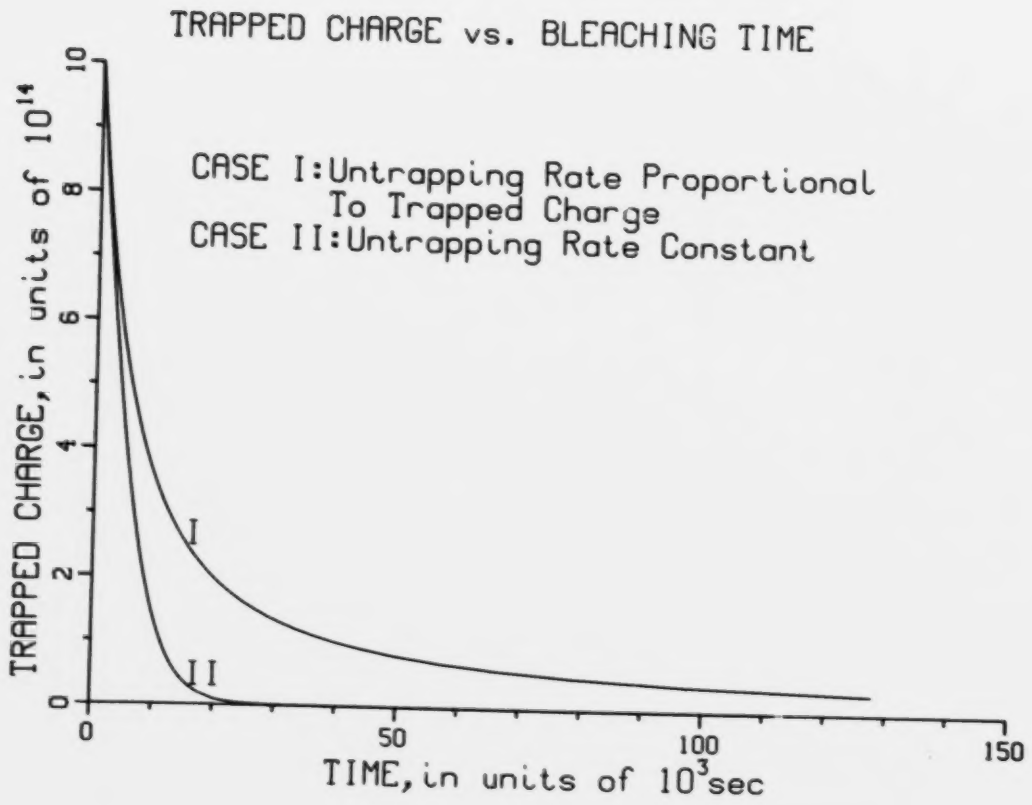

Fig. 6:- Trap charge vs. bleaching time curves computed from equations (11) and (13) with $N_1 = 10^{16}$ traps/cm^3, $n_{o1} = 10^{15}$ charges/cm^3, $\sigma_r/\sigma_{t1} = 10.0$, $\phi\sigma_{u1} = 2.78 \times 10^{-4}$ (1.0/hr, i.e. $\phi n_{c1}\sigma_{u1} = 10^{15}$ charges/hr) for case I and $\phi N_c\sigma_c = 2.78 \times 10^{-4}$ charges/sec (10^{15} charges/hr) for case II. As expected case II bleaching proceeds much faster than case I. However, even in this case the trapped charge does not reach zero until the bleaching time is infinite.

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trapped charge vs. bleaching time curve is not exponential, i.e. it is not linear on a semi-log plot. This point will be discussed in the next section. Third, as expected, Case I bleaching proceeds appreciably slower than Case II. This is demonstrated by Fig. 6, which shows both cases, i.e. equations (11) and (13), computed for the same set of constants and for equal charge untrapping rates at t = 0.

The bleaching equations obtained by using the retrapping conditions which were applied to derive the usual first and second order TL glow curve expressions: It is useful to determine the bleaching behavior obtained by inserting the conditions used to derive the usual glow curve expressions, equations (7) and (9), into the bleaching equations, (11) and (13). To begin, the condition $n_1 = n_r$ was used in the derivation of (11) and (13). When the other condition used to obtain the usual second order glow curve expression, namely $\sigma_{t1} = \sigma_r$ is inserted into (11) one obtains

$$n_1 = \frac{N_1 n_{o1}}{n_{o1}(\phi\sigma_{u1})t + N_1} . \tag{14}$$

Likewise (13) becomes, using the same condition

$$n_1 = n_{o1}\exp(-\phi N_c \sigma_c t/N_1). \tag{15}$$

Thus, if the optical untrapping rate is proportional to the trapped charge, and the other approximations apply, a curve of trapped charge concentration vs. bleaching time should follow (14). However, if the untrapping rate is constant the bleaching rate would follow an exponential decay curve, equation (15).

As discussed above, the usual first order TL expression is obtained by letting $\sigma_r >> \sigma_{t1}$. If (11) is subjected to this condition one obtains

$$n_1 = n_o\exp[-(\phi\sigma_{u1})t]. \tag{16}$$

In other words, if the material contains a single type of electron trap and single type of hole trap, if the bleaching rate is proportional to the trapped charge, and all other conditions apply that lead to first order kinetics, optical bleaching should cause the trapped charge concentration to decay exponentially. Likewise, if (13) is subjected to the $\sigma_r >> \sigma_{t1}$ condition it becomes

$$n_1 = n_o - (\phi N_c \sigma_c)t, \tag{17}$$

i.e. if the bleaching occurs at a constant rate the trapped charge concentration decreases linearly.

Inasmuch as it is highly likely that the untrapping rate is proportional to the trapped charge concentration in most materials, the appearance of

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purely exponential bleaching behavior indicates that the corresponding glow curves should be first order. In cases where retrapping occurs only to a small extent the glow curves would be more like first order than second order.

The general optical bleaching equations for the case of one type of electron trap and one type of hole trap: In the previous section most of the cases considered involve one or more approximations and/or conditions. If none of these approximations apply the bleaching behavior, in the case where the untrapping rate is proportional to the trapped charge conc, is given by the coupled equations

$$\frac{dn_1}{dt} = (-1)(\phi\sigma_{u1}n_1)\left[1 - \frac{\sigma_{t1}(N_1-n_1)}{\sigma_{t1}(N_1-n_1)+\sigma_r n_r}\right] \tag{18}$$

$$\frac{dn_r}{dt} = (-1)(\phi\sigma_{u1}n_1)\left[\frac{\sigma_r n_r}{\sigma_{t1}(N_1-n_1)+\sigma_r n_r}\right]. \tag{19}$$

If the untrapping occurs at a constant rate the same equations apply except that the $(\phi\sigma_{u1}n_1)$ factor is replaced by $(\phi\sigma_c N_c)$. Both sets of these equations have analytical solutions. They very closely resemble the solutions of equations (11) and (13). However, to save space they will not be discussed other than to mention one obvious point. If the initial trapped hole concentration is larger than the initial trapped electron concentration, as the trapped electron concentration approaches zero the trapped hole concentration will approach a finite value which will not diminish with additional bleaching.

V. EFFECTS ON OPTICAL BLEACHING, PRIOR TO TL MEASUREMENT, ON MATERIALS EXHIBITING TWO OR MORE GLOW PEAKS

The changes produced by subjecting samples exhibiting a single glow peak to an increasing amount of optical bleaching prior to TL measurement were described above. To demonstrate that similar changes will occur in materials exhibiting more than one glow peak only two varients of the simplest configuration need be considered. However, the procedure is completely general and is readily extended to systems with any number of different types of electron and hole traps and luminescent emission centers.

Case I--Untrapping proportional to the trapped charge concentration: Consider a system consisting of three types of electron traps with concentrations N_1, N_2, N_3, and one type of hole trap, with conc N_r (N_r will not appear in the equations), which is also a luminescent recombination center. As was done above, assume that only electrons are untrapped by thermal and light induced processes. The probability that an electron is optically untrapped from the N_i type of trap is $\phi\sigma_{ui}n_i$ ($i=1,2,3$) where all symbols are in exact analogy with those used above. Once untrapped electrons are in the conduction band they can, in this case, be retrapped by any of the three types of electron traps that do not contain charges, or alternatively they can enter into recombination with a trapped hole. The total number of processes that the mobile electron can enter into is proportional to

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$$(AP) = \sigma_{t1}(N_1-n_1) + \sigma_{t2}(N_2-n_2) + \sigma_{t3}(N_3-n_3) + \sigma_r n_r . \qquad (21)$$

The fraction of mobile charges retrapped in the ith type of trap is $\sigma_{ti}(N_i-n_i)/(AP)$. Also, at any time t, the total number of light induced mobile charges is proportional to

$$U = \sigma_{u1} n_1 \phi + \sigma_{u2} n_2 \phi + \sigma_{u3} n_3 \phi .$$

Then, in exact analogy with equations (18) and (19), the set of equations which described the trapped charge concentration as a function of bleaching time is

$$\begin{aligned} dn_1/dt &= (-1)\sigma_{u1} n_1 \phi + U\sigma_{t1}(N_1-n_1)/(AP) \\ dn_2/dt &= (-1)\sigma_{u2} n_2 \phi + U\sigma_{t2}(N_2-n_2)/(AP) \\ dn_3/dt &= (-1)\sigma_{u3} n_2 \phi + U\sigma_{t3}(N_3-n_2)/(AP) \\ dn_r/dt &= (-1)U\,\sigma_r n_r/(AP) \ . \end{aligned} \qquad (22)$$

Although this set of equation appears to have an analytical solution, its behavior is more conveniently demonstrated by computer obtained numerical solutions. A solution corresponding to the experimental example described in section II is contained in Fig. 7. In this case it was assumed that, when bleaching was initiated, both the N_1 and N_2 traps were partially filled, the N_3 traps were empty, and that the total trapped electron charge was equal to the total trapped hole charge. The computed charge concentrations closely follow the behavior described in the introduction. The charges in the originally populated traps, the N_1, N_2, and N_r traps, decrease monotonically and the charges in the originally empty traps, the N_3 traps, increase to a maximum and then decrease. Also, it can be shown, although it is intuitively obvious, that the total trapped charge will not reach zero until the bleaching time approaches infinity.

Case II--total charge release rate constant: In analogy with the "slowest" and "fastest" bleaching cases described above, it is useful to consider the three types of electron trap and one type of hole trap configuration under circumstances where the charge is optically excited to the conduction band at a constant rate. However, since there is more than one type of trapping center, the light released charges will arise from all three types of electron traps and the release rate for the ith type of trap will be given by

$$(\phi N_c \sigma_c)[(n_i v_{u1})/(n_i v_{u2} + n_2 v_{u2} + n_3 v_{u3})] . \qquad (23)$$

Here v_{ui} is written instead of σ_{ui} to provide for the possibility that the relative untrapping cross sections may be different from those applying to direct photoexcitation. Using these definitions and analogs of those used

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above, the bleaching equations for this case become

$$\frac{dn_i}{dt} = (-1)(\phi N_c \sigma_c)\left[\frac{n_i v_{ui}}{n_1 v_{u1} + n_2 v_{u2} + n_3 v_{u3}} - \frac{\sigma_{t1}(N_i - n_i)}{(AP)}\right]$$

where i = 1,2,3 and

$$\frac{dn_r}{dt} = (-1)(\phi N_c \sigma_c)\left[\frac{n_r \sigma_r}{(AP)}\right]. \qquad (24)$$

A computer obtained numerical solution of this equation set is shown in Fig. 8 for constants that make it directly comparable to Fig. 7. (Note the scale change.) Except for the expected fact that the trapped charge concentration diminishes at a faster rate it reproduces all of the features contained in Fig. 7.

Thus, it is reasonable to conclude that the optical bleaching behavior described in this section, and summarized in Figs. 7 and 8, will occur in materials in which light or thermally released trapped charges have a non-negligible probability of undergoing retrapping. This point could be emphasized by computing solutions for equations (22) and (24) for successively smaller values of the retrapping cross sections and/or successively larger values of the recombination cross section. Actually, this conclusion can be obtained by inspection of (22) and (24).

Lastly, it should be pointed out that the changes in trapped charge concentration which occur as a result of optical bleaching, particularly those demonstrated in Figs. 7 and 8, correspond well with the optical bleaching measurements of Wintle and Huntley (1979, 1980) which were described in Section II.

VI. SUMMARY

The preceding text is the basis for numerous conclusions, of which only the most important are listed here. First, a number of different studies exist which can be understood on the basis that the materials investigated exhibit characteristics indicative of non-negligible charge retrapping during bleaching or TL measurements. Second, as can be obtained from the original publications, the usual first order glow curve expression will describe glow peak shapes precisely only when retrapping is negligible, i.e. when the retrapping cross sections are negligible and/or the recombination cross sections are very large. Third, the usual second order glow curve expression will describe glow peaks precisely only when the trapped electron and hole concentrations are equal and the cross sections for retrapping and recombination are equal. This suggests that only the highest temperature glow peak in a glow curve containing more than one peak, in a material containing only one type of recombination center, should be describable by the usual kinetic expression. Fourth, when retrapping occurs bleaching curves should not be

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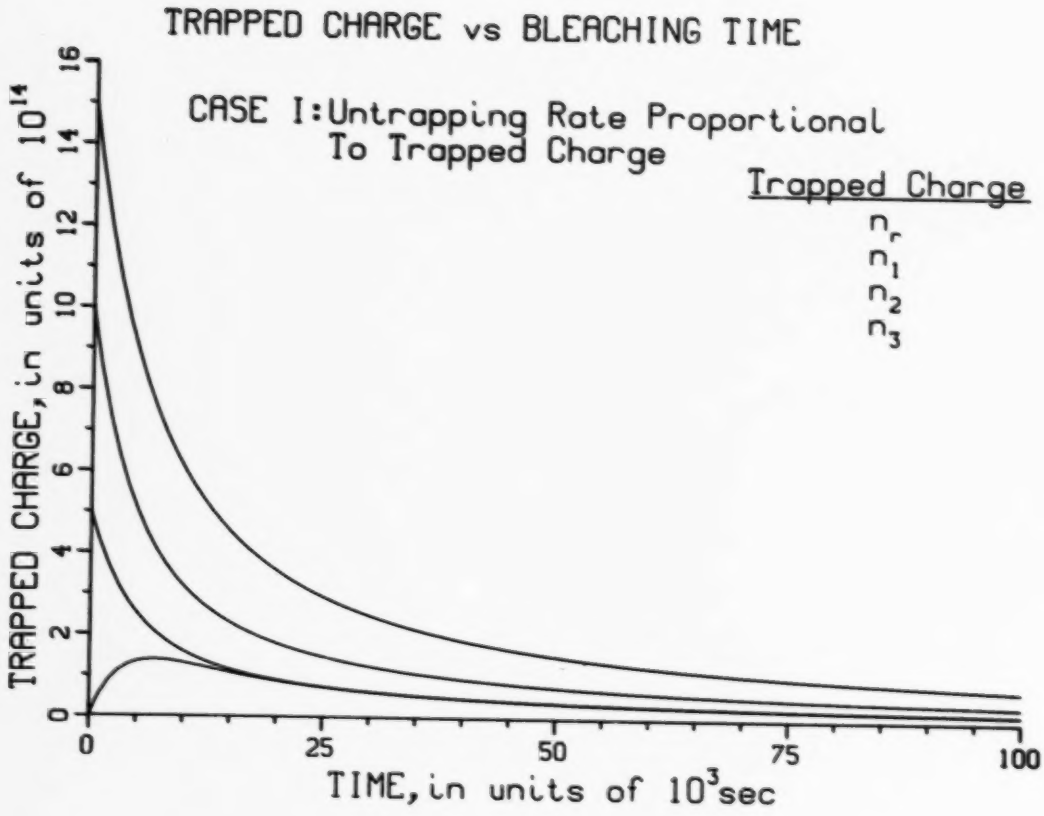

Fig. 7:- Trapped charge vs. bleaching time curves computed from (22) using the parameters $N_1 = 0.5$, $N_2 = 1.0$, $N_3 = 0.5 \times 10^{16}$ traps/cm^3, $n_{o1} = 10^{15}$, $n_{o2} = 5 \times 10^{14}$, $n_{o3} = 0$, $n_{ro} = 1.5 \times 10^{15}$ charges/cm^3, $\sigma_r/\sigma_{t1} = 10$ and $\phi\sigma_{uj} = 2.78 \times 10^{-4}$/sec (1.0/hr), i.e. $\phi(\sigma_{u1}n_{o1} + \sigma_{u2}n_{o2}) = 1.5 \times 10^{15}$ charges/hr. These curves contain all of the features exhibited by the bleaching measurements on ocean sediments described in section II.

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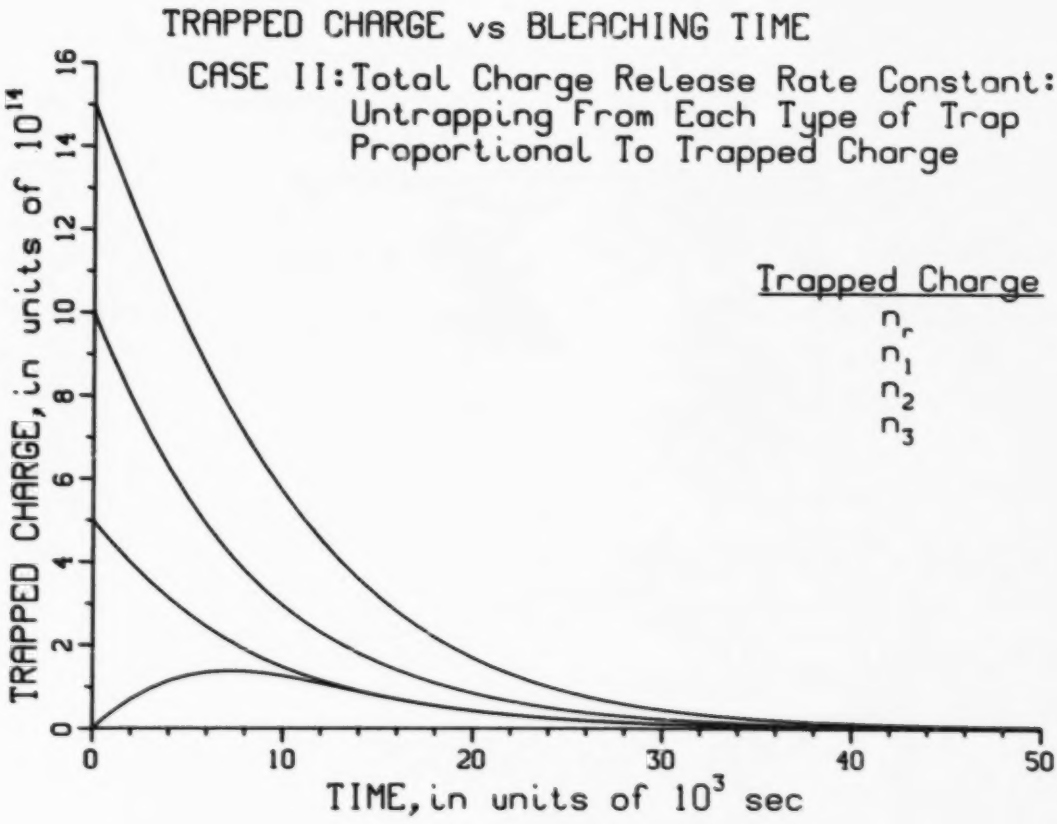

Fig. 8:- Trapped charge vs. bleaching time curves computed from (24) with the same parameters used for Fig. 7, except for $\phi N_c \sigma_c = 4.17 \times 10^{11}$ charges/sec (1.5×10^{15}/hr) and $v_r/v_{ti} = 10$. These curves show all of the features contained in Fig. 7 except that the trapped charge concentration diminishes much more rapidly.

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precisely exponential. Fifth, bleaching prior to TL measurement can cause some glow peaks to decrease and others to first increase and then decrease. Sixth, when retrapping occurs bleaching cannot reduce the trapped charge to zero in a finite time.

To conclude, this paper does not describe all of the consequences of charge retrapping on TL measurements. For example, the effects of retrapping on glow curves containing more than one glow peak will be described in a separate paper.

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